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IS 12308-10 (1991): Methods for Chemical Analysis of Cast Iron and Pig Iron, Part 10: determination of managanese (upto 7.0 percent) by arsenite(volumetric)method [MTD 6: Pig iron and Cast Iron]



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भारतीय मानक

ढलवाँ लोहे एवं कच्चे लोहे का रासायनिक विश्लेषण – पद्धतियाँ

भाग 10 आर्सेनाइट, आयतनी पद्धति से मैंगनीज (7.0 प्रतिशत तक) ज्ञात करना

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 10 DETERMINATION OF MANGANESE (UP TO 7.0 PERCENT)
BY ARSENITE (VOLUMETRIC) METHOD

UDC 669.162.275.12 + 669.13 + 543.24 [546.711]

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Part 10) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Chemical analysis of cast iron and pig iron was covered in IS 228 : 1959 'Methods of chemical analysis of pig iron, cast iron and plain carbon and low alloy steels (revised)'. During its second revision it was decided that a comprehensive series should be prepared for chemical analysis of all types of steels and the other covering the chemical analysis of cast iron and pig iron. Accordingly IS 228 on revision was published in several parts covering chemical analysis of various steels only and a separate series of standards under IS 12308 is being published for chemical analysis of cast iron and pig iron. This standard (Part 10) is one in the latter series. The other parts in the series are :

IS 12308 Methods for chemical analysis of cast iron and pig iron :

Part 1 Determination of total carbon by thermal conductivity method

Part 2 Determination of sulphur by iodimetric titration method

Part 3 Determination of manganese by periodate spectrophotometric method

Part 4 Determination of total carbon, graphitic carbon and combined carbon by gravimetric method

Part 5 Determination of phosphorus by alkalimetric method (for phosphorus 0.01 to 0.50 percent)

Part 6 Determination of silicon by gravimetric method (for silicon 0.1 to 6.0 percent)

Part 7 Determination of nickel by dimethylglyoxime (Gravimetric) method (for nickel 0.5 to 36 percent)

Part 8 Determination of chromium by persulphate (oxidation) method (for chromium 0.1 to 28 percent)

Part 9 Determination of molybdenum by thiocyanate (Spectrophotometric) method (for molybdenum 0.1 to 1.0 percent)

Part 10 Determination of total carbon by the direct combustion (volumetric) method (for carbon 1.50 to 4.50 percent)

In this part the bismuthate method for determination of manganese (in the absence of cobalt) in cast iron and pig iron has been replaced by arsenite volumetric method for determination of manganese (in absence of cobalt) in plain cast iron and pig iron.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF CAST IRON AND PIG IRON

PART 10 DETERMINATION OF MANGANESE (UP TO 7.0 PERCENT) BY ARSENITE (VOLUMETRIC) METHOD

1 SCOPE

1.1 This standard (Part 10) prescribes the method for determination of manganese (up to 7.0 per cent) in plain cast iron and pig iron containing less than 0.5 per cent of chromium and no cobalt content.

1.2 This method is not applicable to alloy cast iron.

2 SAMPLING

The sample shall be drawn and prepared as prescribed in the relevant Indian Standard.

3 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

4 METHOD

4.1 Outline of the Method

The sample is dissolved in acid mixture (phosphoric acid, sulphuric acid and nitric acid). Manganese is oxidised by ammonium persulphate in the presence of silver nitrate and titrated with standard sodium arsenite solution.

4.2 Reagents

4.2.1 Acid Mixture

To 400 ml of water, cautiously, add 100 ml of sulphuric acid ($rd=1.84$), 125 ml of phosphoric acid ($rd=1.75$) and 250 ml of nitric acid ($rd=1.42$). Cool, dilute to 1 litre and mix.

4.2.2 Silver Nitrate Solution

0.2 percent (m/v).

4.2.3 Sodium Chloride Solution

0.2 percent (m/v).

4.2.4 Ammonium Persulphate Solution

10 percent (m/v), freshly prepared.

4.2.5 Standard Iron

having same manganese content as the sample.

4.2.6 Sodium Arsenite ($NaAsO_2$)

solid.

4.2.6.1 Standard arsenite solution

Dissolve 1.05 g of sodium arsenite in 2 litres of water containing 2 g of sodium bicarbonate. Standardise this solution (see 4.3) with an iron sample of known manganese (4.2.5) content.

4.3 Procedure

4.3.1 Transfer 0.100 g sample to a 250-ml conical flask. Add 30-ml of acid mixture. Heat gently to dissolve the sample and boil off nitrous fumes. Dilute to 50-ml with water, filter through a medium textured filter paper and wash 3-4 times with hot water. To the filtrate, add 10 ml of silver nitrate solution, followed by 20 ml of ammonium persulphate solution. Boil for 20 s, remove from hot plate and cool rapidly to room temperature. Add 10 ml of sodium chloride solution and titrate immediately with standard sodium arsenite solution shaking vigorously until the pink colour is just discharged.

Take 0.100 g of a standard iron sample of a known manganese content and proceed as above. Both standard and sample are to be titrated under identical operative conditions.

NOTE—It is necessary to use standard iron sample having same manganese content as the sample under analysis.

4.3.2 Run a blank using all the reagents, through the procedure specified in 4.3.1.

4.4 Calculation

Manganese, percent by mass =

$$\frac{V_1 - V_2}{V_3 - V_2} \times \text{percentage of manganese in the standard}$$

where

V_1 = volume, in ml, of sodium arsenite used for sample;

V_3 = volume, in ml, of sodium arsenite used for standard; and

V_2 = volume, in ml, of sodium arsenite used for blank.

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